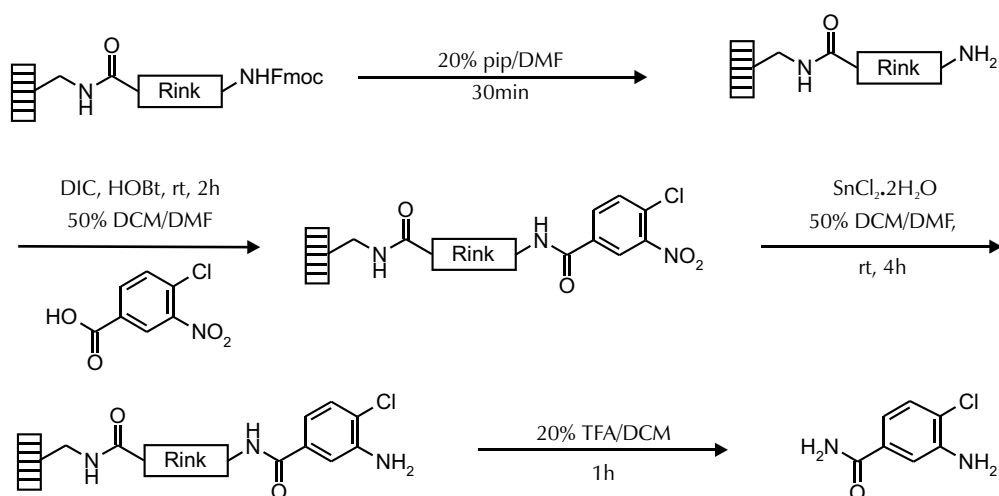


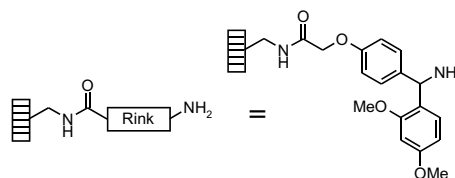


Reduction of a Support-Bound Aryl Nitro Group

Support-bound nitrobenzenes can be reduced to the corresponding anilines by tin(II) chloride dihydrate. In the following example, the nitrobenzene is attached to the Rink amide linker on SynPhase™ PS Lanterns.¹



DCM: dichloromethane
DMF: dimethylformamide
pip: piperidine
HOBt: hydroxybenzotriazole
DIC: diisopropylcarbodiimide
rt: room temperature
TFA: trifluoroacetic acid



Fmoc Deprotection

D-Series Lanterns are covered with 20% pip/DMF and stood at rt for 30min. The solution

is decanted and the Lanterns are washed with DMF (3×3min) and DCM (3×3min).

Benzoic Acid Coupling

Each Fmoc deprotected Lantern (loading: 34μmol) is treated with 0.5mL of a solution of DIC (0.2M, 100μmol, 2.9 mole equivalents), HOBt (0.2M, 100μmol, 2.9 mole equivalents) and 4-chloro-3-nitrobenzoic acid (0.2M,

100μmol, 2.9 mole equivalents) in 50% DCM/DMF at rt for 2h. The solution is decanted and the Lanterns are washed with DMF (3×3min) and DCM (3×3min).

Reduction

Each **D-Series Lantern** is treated with 0.5mL of a 2M solution of tin(II) chloride dihydrate (1000 μmol , 29 mole equivalents) in 50% DCM/DMF at

rt for 4h. The solution is decanted and the Lanterns are washed with DMF (3 \times 3min) then DCM (3 \times 3min).

Cleavage

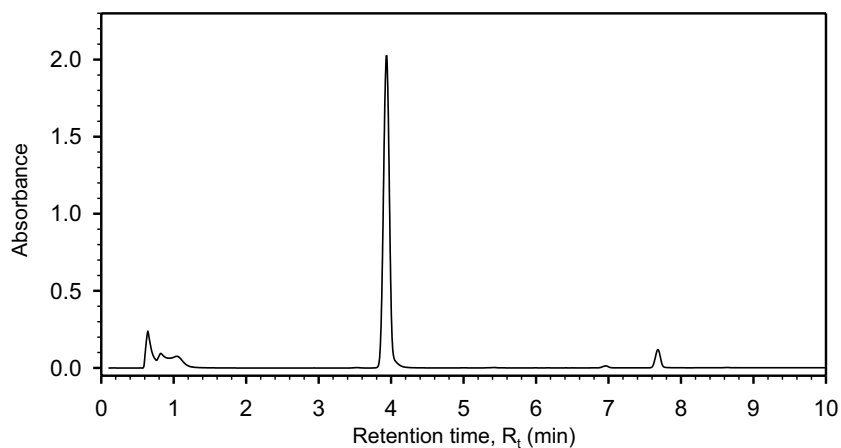
Individual Lanterns are placed in polypropylene tubes and treated with 20% TFA/DCM (0.6-0.8mL) for 1h. The Lanterns are removed and the cleaved

products are concentrated using a centrifugal evaporator. Samples are dissolved in 90% $\text{CH}_3\text{CN}/\text{H}_2\text{O}$ for HPLC and ES-MS analysis.

Analytical Data

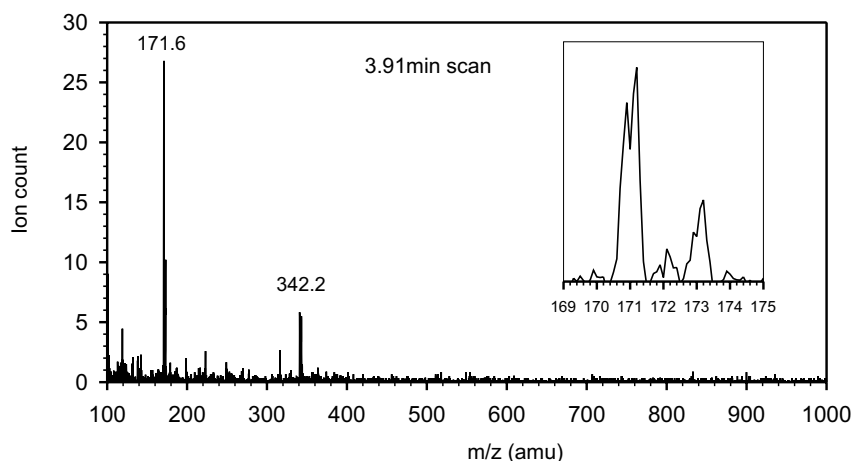
Reverse-phase HPLC trace of the crude aniline

Detection at 214nm



Electrospray MS trace of LC peak at $R_t = 3.91\text{ min}$

Molecular Formula: $\text{C}_7\text{H}_7\text{ClN}_2\text{O}$
Monoisotopic Mol. Weight: 170.6amu
 $[\text{M}+\text{H}]^+ = 171.6\text{amu}$
 $[2\text{M}+\text{H}]^+ = 342.2\text{amu}$



Note

1 The chemistry described here was performed using SynPhase PS Lanterns but is readily adaptable to SynPhase PA Lanterns.



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